PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶:
B01J 10/00, C02F 1/02, C07C 273/04

1 (11) 1110111

(11) International Publication Number:

WO 97/15388

A1

(43) International Publication Date:

1 May 1997 (01.05.97)

(21) International Application Number:

PCT/IB96/01004

(22) International Filing Date:

27 September 1996 (27.09.96)

(30) Priority Data:

3033/95

27 October 1995 (27.10.95)

CH

(71) Applicant (for all designated States except US): UREA CASALE S.A. [CH/CH]; Via Sorengo 7, CH-6900 Lugano-Besso (CH).

(72) Inventors; and

(75) Inventors/Applicants (for US only): PAGANI, Giorgio [IT/IT]; Viale Facaza, 26/6, I-20142 Milano (FT). ZARDI, Federico [IT/CH]; Via Lucino 60, CH-6932 Breganzona (CH).

(74) Agent: ZARDI, Marco; Via Sorengo 7, CH-6900 Lugano-Besso (CH). (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

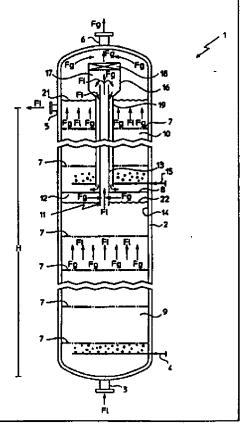
Published

With international search report.

(54) Title: HYDROLYSIS REACTOR FOR REMOVAL OF UREA, AMMONIA AND CARBON DIOXIDE FROM A LIQUID PHASE COMPRISING UREA IN AQUEOUS SOLUTION

(57) Abstract

A hydrolysis reactor for removal of urea, ammonia and carbon dioxide from a liquid phase comprising urea in aqueous solution advantageously, advantageously comprises: a dividing baffle (8) extending horizontally at a preset height in the reactor, in which it defines a first and a second respectively lower and upper reaction space (9, 10); means (11) of collection and extraction from the reactor of a first portion of a gaseous phase comprising high pressure and temperature steam, previously fed into the first reaction space (9); and means (15) for feeding a second portion of the gaseous phase comprising high pressure and temperature steam to the second reaction space (10).



FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

| AM | Armenia | GB | United Kingdom | MW | Malawi |
|----|--------------------------|----|------------------------------|----|--------------------------|
| AT | Austria | GE | Georgia | MX | Mexico |
| AU | Australia | GN | Guinea | NE | Niger |
| BB | Barbados | GR | Greece | NL | Netherlands |
| BE | Belgium | HÜ | Hungary | NO | Norway |
| BF | Burkina Faso | IE | Ireland | NZ | New Zealand |
| BG | Bulgaria | ľT | Italy | PL | Poland |
| BJ | Benin | JP | Japan | PT | Portugal |
| BR | Brazil | KE | Kenya | RO | Romania |
| BY | Belanus | KG | Kyrgystan | ŔŨ | Russian Federation |
| CA | Canada | KP | Democratic People's Republic | SD | Sudan |
| CF | Central African Republic | | of Korea | SE | Sweden |
| CG | Congo | KR | Republic of Korea | SG | Singapore |
| CH | Switzerland | KZ | Kazakhstan | SI | Slovenia |
| ĊI | Côte d'Ivoire | u | Liechtenstein | SK | Slovakia |
| CM | Cameroon | LK | Sri Lanka | SN | Senegal |
| CN | China. | LR | Liberia | SZ | Swaziland |
| CS | Czechoslovakia | LT | Lithuania | TD | Chad |
| CZ | Czech Republic | LU | Luzembourg | TĢ | Togo |
| DĒ | Germany | LV | Latvia | TJ | Tajikistan |
| DK | Denmark | MC | Monaco | TT | Trinidad and Tobago |
| EE | Estonia | MD | Republic of Moldova | UA | Ukraine |
| ES | Spain | MG | Madagascar | UĢ | Uganda |
| FI | Finland. | ML | Mali | us | United States of America |
| FR | France | MN | Mongolia | υz | Uzbekistan |
| GA | Gabon | MR | Mauritapia | VN | Vict Nam |

"Hydrolysis reactor for removal of urea, ammonia and carbon dioxide from a liquid phase comprising urea in aqueous solution"

DESCRIPTION

5 Field of the Invention

The present invention relates to a hydrolysis reactor for removal of urea, ammonia and carbon dioxide from a liquid phase comprising urea in aqueous solution.

As known, waste waters resulting from the purification and recovery process of urea produced in a high-pressure and temperature synthesis reactor have a high residual urea content in aqueous solution generally between 500 ppm and 30'000 ppm and as such cannot be freely discharged into the environment because of the severe antipollution laws in force in industrialized countries.

Each urea production plant must therefore provide appropriate equipment capable of removing residual urea from the waste water so as to lower its concentration to a few ppm, preferably less than 10 ppm.

In the field of treatment of waste waters resulting from the urea purification and recovery process, the requirement for making available residual urea hydrolysis reactors, which would allow on the one hand to obtain non-polluting waste waters with a urea content less than 10 ppm and on the other hand to recover the residual urea (in the form of ammonia and carbon dioxide) contained in these waste waters fed to the hydrolysis reactor, is thus strongly felt.

Prior Art

In order to meet the above mentioned requirement, vertical 30 hydrolysis reactors in which flows a liquid phase comprising urea in aqueous solution and a gaseous phase

- 2 -

comprising high pressure and temperature steam, generally between 15 bar and 30 bar and between 150°C and 250°C, have been becoming more widely used.

These reactors contain a plurality of horizontally extending perforated plates.

5

10

The perforated plates have the function of facilitating the mutual mixing of the phases to encourage intimate contact, and thus exchange of mass and heat essential for the hydrolysis reaction of the urea in ammonia (NH_3) and carbon dioxide (CO_2) , and for simultaneous extraction of the NH_3 and CO_2 from the liquid phase to the gaseous phase.

The heat necessary for decomposition of the urea and extraction of the reaction products from the liquid phase is supplied by the steam contained in the gaseous phase.

- 15 Canadian patent application CA-A-2 141 886 describes a reactor of this type in which the liquid phase and the gaseous phase are made to flow in co-current from below upwards through a plurality of horizontal perforated plates.
- Although advantageous in many ways, the hydrolysis reactor described above exhibits a number of drawbacks, the first of which is that to obtain the desired degree of decomposition of the urea and related extraction from the liquid phase of the NH₃ and the CO₂ produced, it is necessary to operate with excess of steam to prevent the hydrolysis reaction from reaching the equilibrium before its time and the gaseous solution from becoming saturated with the reaction products already during its passage through the reactor.
- 30 As a result, to obtain an aqueous solution with a residual urea content lower than 10 ppm it is necessary to use high quantities of high pressure and temperature steam, with the ensuing high energy and steam consumption and high

operation costs.

Summary of the Invention

The technical problem underlying the present invention is to make available a hydrolysis reactor for removal of urea, ammonia and carbon dioxide from a liquid phase comprising urea in aqueous solution, which would permit operation with low steam and energy consumption and low operating costs and allow at the same time to obtain a liquid phase with a urea content below 10 ppm.

- 10 In accordance with a first embodiment of the present invention, the above mentioned problem is solved by a hydrolysis reactor for removal of urea, ammonia and carbon dioxide from a liquid phase comprising urea in aqueous solution, comprising:
- 15 a substantially cylindrical vertical external shell;
 - a plurality of perforated plates superimposed and extending horizontally and in mutually spaced relationship in said shell;
- an inlet opening for said liquid phase arranged proximate
 to a lower end of said shell;
 - first means of supplying a first portion of a gaseous phase comprising high pressure and temperature steam, supported in said shell above said liquid phase inlet opening;
- 25 an outlet opening for said liquid phase arranged proximate to an upper end of said shell;
 - an outlet opening for said gaseous phase arranged proximate to the upper end of said shell;

and characterized in that it comprises:

WO 97/15388

10

15

20

- 4 -

- a dividing baffle extending horizontally at a preset height in said shell, in which it defines a first and a second respectively lower and upper reaction space;
- means of collection and extraction from the shell of said
 first gaseous phase portion, supported proximate to said
 dividing baffle in said first reaction space;
 - second means of feeding a second portion of said gaseous phase comprising high pressure and temperature steam, supported above said dividing baffle in said second reaction space.

Advantageously, in the hydrolysis reactor according to the present invention, the reaction space in the shell is appropriately divided by a dividing baffle in a first and a second reaction space each of which is fed with a respective gaseous phase portion comprising steam.

In this manner it is possible to use efficiently and rationally the high pressure and temperature steam necessary for decomposition of the urea and extraction of the reaction products, so as to obtain, for equal liquid phase purification degree, a substantial reduction of the quantity of steam to be fed into the hydrolysis reactor with respect to prior art reactors.

Indeed, thanks to the present invention, purification of the liquid phase takes place appropriately in two distinct reaction spaces, into each of which is fed the quantity of steam strictly necessary for obtaining a liquid phase outlet from the hydrolysis reactor with a residual urea concentration below 10 ppm.

In particular, extraction from the first reaction space of the gaseous phase now saturated with reaction products and feeding into the second reaction space of a new gaseous phase comprising high pressure and temperature steam, permit hydrolysis also of the last traces of urea contained

30

in the liquid phase as well as recovery of the NH₃ and CO₂ in the gaseous phase without thereby having to employ excess of steam.

The liquid phase thus purified can be discharged into the environment but can also be advantageously reused as high temperature and pressure water in the urea synthesis plant or for other industrial uses, e.g. as boiler water.

Another advantage of the hydrolysis reactor which is the object of the present invention lies in the fact that, for equal residual urea concentration contained in the outlet liquid phase, the residence time of the liquid phase in the reactor is significantly lower than the residence time in the prior art reactors.

This permits building a hydrolysis reactor with dimensions and investment costs considerably less than those of the prior art.

Particularly advantageous results were found by arranging the dividing baffle at a height between 55% and 80% of the useful height of the shell.

- In the description given below and in the following claims, the term "useful height", is understood to mean the height of the shell usable for the urea hydrolysis reaction. In this particular case the useful height is defined by the level reached by the liquid phase in the shell.
- 25 Preferably, the dividing baffle is arranged at a height between 65% and 75% of the useful shell height.

In this manner, there is obtained with small amounts of high pressure and temperature steam and low operating costs, a concentration of urea in liquid phase in the first reaction space generally between 30 ppm and 70 ppm and in the second reaction space between 0 ppm and 5 ppm.

PCT/IB96/01004 WO 97/15388 - 6 -

In accordance with this first embodiment of the present invention, the hydrolysis reactor includes advantageously a dividing baffle extending horizontally for substantially the entire cross section of the shell.

- In addition, the collection and extraction means comprise advantageously:
 - a collection chamber for the first gaseous phase portion, formed in the first reaction space between the dividing baffle and an inner wall of the shell;
- a duct extending coaxially in the shell between the 10 collection chamber and the gaseous phase outlet opening, for extraction from the first reaction space of a two-phase gas/liquid flow.
- As a result, practical implementation of the present invention is simple in construction and of low realization 15 cost.

the hydrolysis reactor comprises in Advantageously, qas/liquid separator placed between the addition a extraction duct and the gaseous phase outlet opening, so 20 that the liquid phase if any entrained in the gaseous phase leaving the reactor can be recycled to the reactor and thus permit recovery of the urea contained therein.

In accordance with a second embodiment of the present invention, the above mentioned problem is also solved by a hydrolysis reactor for removal of urea, ammonia and carbon dioxide from a liquid phase comprising urea in aqueous solution, comprising:

- a substantially cylindrical vertical external shell;

25

- a plurality of perforated superimposed plates extending horizontal and in mutually spaced relationship in the 30 shell:

- an inlet opening of said liquid phase arranged proximate to a lower end of said shell;
- first means for feeding a first portion of a gaseous phase comprising high pressure and temperature steam, supported in said shell above said liquid phase inlet opening;
 - an outlet opening for said gaseous phase arranged proximate to an upper end of the shell;

and characterized in that it comprises:

25

- a dividing baffle extending horizontally in the shell at a preset height and for substantially the entire cross section of the shell, with the baffle defining in said shell a first and a second respectively lower and upper reaction space;
- 15 a collection chamber for said first gaseous phase portion, formed in said first reaction space between said dividing baffle and an inner wall of said shell;
- a duct extending coaxially in the shell between said collection chamber and said gaseous phase outlet opening, for extraction from said first reaction space of a two-20 phase gas/liquid flow;
 - second means of feeding a second portion of said gaseous phase comprising high pressure and temperature steam, supported above said dividing baffle in said second reaction space;
 - an outlet opening for said liquid phase arranged in said second reaction space proximate to and above said dividing baffle.
- Advantageously, in this embodiment of the present invention the liquid phase and the gaseous phase are made to flow in 30 counter-current in the second reaction space.

WO 97/15388 PCT/IB96/01004

In this manner, it is possible to further improve the mixing of the phases and hence mass and heat exchange so as to facilitate the urea hydrolysis reaction and absorption of the reaction products by the steam.

The characteristics and advantages of the hydrolysis reactor according to the invention are set forth in the description of an embodiment thereof given below by way of non-limiting example with reference to the annexed drawings.

10 Brief description of the drawings

In the drawings:

- FIG. 1 shows a longitudinal cross section of a urea hydrolysis reactor according to a first embodiment of the present invention;
- 15 FIG. 2 shows a longitudinal cross section of a urea hydrolysis reactor according to a second embodiment of the present invention, and
 - FIG. 3 shows a longitudinal cross section in enlarged scale of a detail of the hydrolysis reactor of FIG. 1.

20 Detailed description of a preferred embodiment

With reference to FIGS. 1 and 2, reference number 1 indicates as a whole a hydrolysis reactor for removal of urea, ammonia and carbon dioxide from a liquid phase comprising urea in aqueous solution.

- 25 The reactor 1 comprises a substantially cylindrical vertical external shell 2, provided at its lower end with an inlet opening 3 for a liquid phase comprising urea in aqueous solution, and with means 4 for feeding a first portion of a gaseous phase comprising high pressure and
- 30 temperature steam.

10

15

20

The shell 2 also has a liquid phase outlet opening 5 and a gaseous phase outlet opening 6 arranged at an upper end of the reactor.

A plurality of superimposed perforated plates, all indicated by reference number 7, extend horizontally and in mutually spaced relationship in the shell 2.

The plates 7 are homogeneously distributed along the useful height H of the shell, and are provided with appropriate passages for the liquid phase and the gaseous phase to facilitate mixing of the phases.

FIG. 3 shows in enlarged scale a detail of a perforated plate 7, provided according to a particularly advantageous and preferred embodiment of the present invention, which can be installed into the hydrolysis reactor shown in FIG. 1, as well as in the reactor of FIG. 2.

The perforated plate 7 comprises a plurality of elements 71 with substantially trapezoid cross section, defining within them respective cavities 72, as well as appropriate openings 73 and 74 for passage of the liquid and gaseous phases respectively.

Alternatively, the element 71 can have a substantially rectangular cross section.

The gaseous phase passage openings 74 are defined in an upper wall 75 of the elements 71.

25 The liquid phase passage openings 73 are defined in a side wall 76 of the elements 71.

Advantageously the liquid phase passage openings 73 have a size greater than that of the openings 74 for passage of the gaseous phase.

30 In the description given below and in the following claims, the term "size", is understood to mean the cross section

WO 97/15388 PCT/IB9

area of an opening.

15

30

In the cavity 72 proximate to the wall 75 there is advantageously defined a gaseous phase collection zone 77.

Thanks to this particular structure of the plates, the gaseous phase is separated from the liquid phase during the passage through the plates, to then be again mixed with the liquid phase in finely distributed form.

In this manner it is possible to obtain a significant improvement in the mixing of the phases during their passage through the hydrolysis reactor.

This involves another reduction of the quantity of steam necessary for performance of the hydrolysis and extraction of the reaction products, as well as of the residence time of the liquid phase in the reactor, with resulting reduction of the reactor dimension, of the steam and energy consumption and of the operating and investment costs compared to the prior art reactors.

Plates of this type are described for instance in US-A-5 304 353, with reference to a urea synthesis reactor.

20 Reference number 8 indicates a dividing baffle extending horizontally at a predetermined height of the shell 2. The baffle 8 defines in the shell a first reaction space 9 and a second reaction space 10, respectively lower and upper.

Advantageously, the dividing baffle 8 extends horizontally for substantially the entire cross section of the shell 2.

In the examples of FIGS. 1 and 2, significantly reduced consumption of high pressure and temperature steam is achieved by arranging the dividing baffle 8 at a height corresponding to approximately 70% of the useful height H of the shell 2.

Means 11 for collection and extraction from the shell 2 of

the first gaseous phase portion are supported proximate to the dividing baffle 8 in the first reaction space 9.

The means 11 comprise advantageously a chamber 12 for collection of the first gaseous phase portion and a duct 13 for extraction from the first reaction space 9 of a two-phase gas/liquid flow.

The collection chamber 12 is formed in the first reaction space 9 between the dividing baffle 8 and an inner wall 14 of the shell 2.

The extraction duct 13 is equipped with respective gaseous and liquid phase inlet openings and extends coaxially in the shell 2, between the collection chamber 12 and the gaseous phase outlet opening 6.

A second portion of the gaseous phase comprising high pressure and temperature steam is fed to the reactor by appropriate means 15 supported above the dividing baffle 8 in the second reaction space 10.

Preferably, the feeding means 4 and 15 for the gaseous phase are the type comprising a feed duct connected to a gas distributor in the shell 2. These means are generally known and commonly used in prior art hydrolysis reactors.

20

25

With reference to FIG. 1, the liquid phase outlet opening 5 is arranged in the second reaction space 10 proximate to the upper end of the shell 2. In the example of FIG. 2, the liquid phase outlet opening 5 is arranged in the second reaction space 10 proximate to and above the dividing baffle 8.

As shown in FIGS. 1 and 2, the hydrolysis reactor according to the present invention comprises advantageously a gas/liquid separator 16 placed between the extraction duct 13 and the gaseous phase outlet opening 6.

PCT/IB96/01004 WO 97/15388 - 12 -

The gas/liquid separator 16 is of the type comprising a chamber 17, coaxial with the extraction duct 13, for separation of the liquid phase from the gaseous phase and a demister 18 for separation of the residual liquid phase contained in the gaseous phase leaving the chamber 17.

In the example of FIG. 1, the liquid phase obtained in the gas/liquid separator 16 is advantageously recycled to the second reaction space 10 by means of a recycling duct 19 extending outside and coaxially with the extraction duct 13 between the separation chamber 17 and the dividing baffle 8. In the example of FIG. 2, the recycling takes place through a liquid passage 20.

In FIGS. 1 and 2, the arrows Fl and Fg indicate the various paths inside the hydrolysis reactor of the liquid phase comprising urea in aqueous solution and of the gaseous phase comprising high pressure and temperature steam respectively.

15

20

30

Reference number 21 also indicates the highest level reached by the liquid phase in the shell 2, while 22 indicates the liquid phase level in the collection chamber 12.

Operation of the hydrolysis reactor according to the present invention is as follows.

With reference to FIG. 1, a liquid phase comprising urea in aqueous solution is fed to the reactor 1 through the inlet opening 3, and caused to flow in co-current from below upward with a first gaseous phase portion comprising highpressure (20-25 bar) and high-temperature (200-220°C) steam along a first reaction space 9 in the shell 2. The gaseous phase is fed to the reactor 1 through the feeding means 4.

In the reaction space 9, the liquid phase and the gaseous phase are mixed together while passing through the perforated plates 7, so that part of the urea present in - 13 -

aqueous solution is hydrolyzed and the resulting reaction products (NH3 and CO2) are extracted from the steam present in the gaseous phase. In this first reaction space, the concentration of urea in the liquid phase fed to the reactor is advantageously lowered to a value generally comprised between 40 ppm and 50 ppm.

Proximate to the dividing baffle 8, the liquid phase and first gaseous phase portion are advantageously collected in the chamber 12 and conveyed by the duct 13 to 10 the separation chamber 17 of the gas/liquid separator 16.

In the chamber 17, the liquid phase coming from the first reaction space 9 is separated from the gaseous phase and recirculated to the second reaction space 10 near the dividing baffle 8 through the duct 19.

The gaseous phase, once separated from the liquid phase in 15 the chamber 17, passes through the demister 18 and leaves the reactor 1 through the outlet opening 6.

In the second reaction space 10, the liquid phase is made to flow (still in co-current) with a second portion of the gaseous phase comprising high pressure and temperature 20 steam.

After passing through the perforated plates 7 and reaching the level 21, the liquid phase leaves the reactor 1 through the outlet opening 5. The urea concentration in the liquid phase coming out of the second reaction space 10 is advantageously less than 10 ppm.

25

30

In turn, the second gaseous phase portion which has traversed the second reaction space 10 and is enriched with NH3 and CO2, leaves the reactor 1 through the outlet opening 6.

With reference to FIG. 2, the liquid phase coming from the first reaction space 9 and separated in the chamber 17 of WO 97/15388 PCT/IB96/01004

the gas/liquid separator 16 is advantageously recycled through the passage 20 to the second reaction space 10 near the level 21.

In accordance with this embodiment of the present invention, the liquid phase flows into the second reaction space 10 from above downward in counter-current with the second gaseous phase portion, to then leave the reactor 1 through the outlet opening 5 placed proximate to the dividing baffle 8.

The hydrolysis reactor according to the present invention operates at a pressure between 15 bar and 25 bar and a temperature between 180°C and 215°C. The residence time of the liquid phase in the first reaction space is preferably between 20 min and 40 min, while in the second reaction space it is preferably between 10 min and 20 min.

EXAMPLE 1

In the following example a comparison is made of the quantity of high pressure and temperature steam necessary to obtain a residual urea concentration in the waste waters below 10 ppm, in the case where a prior art hydrolysis reactor or a hydrolysis reactor according to the various embodiments of the present invention are used. Reference is made to FIGS. 1 and 2.

The hydrolysis reactors considered have the following dimensions.

- Inner shell diameter: 1.5 m

- Useful height: 14.0 m

Operating conditions in the reactor are as follows.

- Pressure: 20 bar

30 - Temperature: 210°C

The reactors contain 10 horizontal perforated plates distributed along the useful height of the cylindrical shell.

In the reactors according to the present invention, the dividing baffle is advantageously arranged at approximately 68% of the useful shell height, between the sixth and seventh perforated plates. For further structural details of these reactors reference is made to FIGS. 1 and 2 and related description.

In the prior art reactor, as in that according to the first embodiment of the present invention (FIG. 1), the liquid and gaseous phases are caused to flow in co-current from below upward through the perforated plates. In the second reaction space of the reactor according to the second embodiment of the present invention (FIG. 2), the liquid phase and the gaseous phase flow in counter-current.

The hydrolysis reactors are fed with 30'000 kg/h of a liquid phase having the following composition.

- NH₃ 10'000 ppm

20 - CO₂ 2'000 ppm

- UREA 10'000 ppm

- H₂O the rest

25

The hydrolysis reactors are also fed with a gaseous phase comprising steam at a pressure of 25 bar and a temperature of 215°C.

To obtain a residual urea concentration of 1 ppm in the liquid phase coming out of the reactor, steam consumption in the various cases is shown below.

In the reactor according to the prior art 30 kg of steam 30 are used for 1'000 kg of liquid phase treated.

WO 97/15388 PCT/IB96/01004

In the reactor of FIG. 1, 22 kg of steam were used for 1'000 kg of liquid phase treated.

In the reactor of FIG. 2, 20 kg of steam were used for 1'000 kg of liquid phase treated.

As may be noted, thanks to the present invention it is possible to achieve a significant steam consumption reduction, equal to approximately 30% of the steam consumption necessary in the prior art hydrolysis reactor. This also results in a substantial lowering of energy consumption and operating costs.

The results of the present example were achieved by means of calculation algorithms available in trade.

***.*.**

From the above discussion there emerge clearly the numerous advantages achieved by the hydrolysis reactor according to the present invention. In particular, there is achieved a reduction in the residual urea concentration contained in the waste waters at values below 10 ppm as well as recovery of the hydrolyzed urea, while operating with low steam and energy consumption and low operating and investment costs.

CLAIMS

- 1. Hydrolysis reactor for removal of urea, ammonia and carbon dioxide from a liquid phase comprising urea in aqueous solution, comprising:
- 5 a substantially cylindrical vertical external shell (2);
 - a plurality of perforated plates (7) superimposed and extending horizontally in mutually spaced relationship within said shell (2);
- an inlet opening (3) for said liquid phase arranged
 proximate to a lower end of said shell (2);
 - first means (4) for feeding a first portion of a gaseous phase comprising high pressure and temperature steam, supported in said shell (2) above said liquid phase inlet opening (3);
- 15 an outlet opening (5) for said liquid phase arranged proximate to an upper end of said shell (2);
 - an outlet opening (6) for said gaseous phase arranged proximate to said upper end of said shell (2);

characterized in that it comprises:

- 20 a dividing baffle (8) extending horizontally at a preset height in said shell (2), in which it defines a first and a second respectively lower and upper reaction space (9,10);
- means (11) of collection and extraction from the shell
 (2) of said first gaseous phase portion, supported
 proximate to said dividing baffle (8) in said first
 reaction space (9;
 - second means (15) for feeding a second portion of said gaseous phase comprising high pressure and temperature steam, supported above said dividing baffle (8) in said

PCT/IB96/01004 WO 97/15388 - 18 -

second reaction space (10).

25

- 2. Reactor according to claim 1, characterized in that said dividing baffle (8) is arranged in said shell (2) at a height between 55% and 80% of the useful height (H) of the shell (2).
- 3. Reactor according to claim 2, characterized in that said dividing baffle (8) is arranged in said shell (2) at a height between 65% and 75% of the useful height (H) of the shell (2).
- 4. Reactor according to claim 1, characterized in that said dividing baffle (8) extends horizontally for substantially the entire cross section of the shell (2), and that said collection and extraction means (11) comprise:
- a collection chamber (12) for said first gaseous phase portion formed in said first reaction space (9) between said dividing baffle (8) and an inner wall (14) of said shell:
- a duct (13) extending coaxially in said shell (2) between said collection chamber (12) and said gaseous phase outlet 20 opening (6) for extraction from said first reaction space (9) of a two-phase gas/liquid flow.
 - 5. Reactor according to claim 4, characterized in that it comprises a gas/liquid separator (16) located between said extraction duct (13) and said gaseous phase outlet opening (6), for separation of the liquid phase from said two-phase flow.
 - 6. Reactor according to claim 5, characterized in that said gas/liquid separator (16) comprises:
- a separation chamber (17) for separating the liquid phase 30 from the gaseous phase, coaxial with said extraction duct (13);

- a demister (18) for separation of the residual liquid phase contained in the gaseous phase coming out of said separation chamber (17);
- a recycling duct (19) extending outside and coaxially with said extraction duct (13) between said separation chamber (17) and said dividing baffle (8), for recycling proximate to and above said dividing baffle (8) the liquid phase obtained in said separator (16).
- 7. Reactor according to claim 1, characterized in that said perforated plates (7) comprise:
 - a plurality of elements (71) with substantially trapezoid or rectangular cross section, defining inside them respective collection zones (77) for said gaseous phase;
- a plurality of openings (73) for passage of said liquid 15 phase defined in association with a side wall (76) of said elements (71);
 - a plurality of openings (74) for passage of said gaseous phase defined in an upper wall of said elements (71) in fluid communication with said collection zones (77);
- with said openings (73) for passage of the liquid phase having a size greater than that of said openings (74) for passage of the gaseous phase.
- 8. Hydrolysis reactor for removal of urea, ammonia and carbon dioxide from a liquid phase comprising urea in aqueous solution; comprising:
 - a substantially cylindrical vertical external shell (2);
 - a plurality of perforated plates (7) superimposed and extending horizontally and in mutually spaced relationship in said shell (2);
- 30 an inlet opening (3) for said liquid phase arranged

WO 97/15388 - 20 -

proximate to a lower end of said shell (2);

- first means (4) for feeding a first portion of a gaseous phase comprising high pressure and temperature steam. supported in said shell 2 above said liquid phase inlet opening (3), and
- an outlet opening (6) for said gaseous phase arranged proximate to an upper end of said shell (2);

characterized in that it comprises:

- a dividing baffle (8) extending horizontally in said 10 shell (2) at a preset height for substantially the entire cross section of the shell (2), with said baffle defining in said shell (2) a first and a second respectively lower . and upper reaction space (9,10);
- a collection chamber (12) for said first gaseous phase portion formed in said first reaction space (9) between said dividing baffle (8) and an inner wall (14) of said shell (2):
- a duct (13) extending coaxially in said shell (2) between said collection chamber (12) and said gaseous phase outlet 20 opening (6), for extraction from said first reaction space (9) of a two-phase gas/liquid flow;
 - second means (15) for feeding a second portion of said gaseous phase comprising high pressure and temperature steam, supported above said dividing baffle (8) in said second reaction space (10);
 - an outlet opening (5) for said liquid phase arranged in said second reaction space (10) proximate to and above said dividing baffle (8).
- 9. Reactor according to claim 8, characterized in that said dividing baffle (8) is arranged in said shell (2) at a height between 55% and 80% of the useful height (H) of the

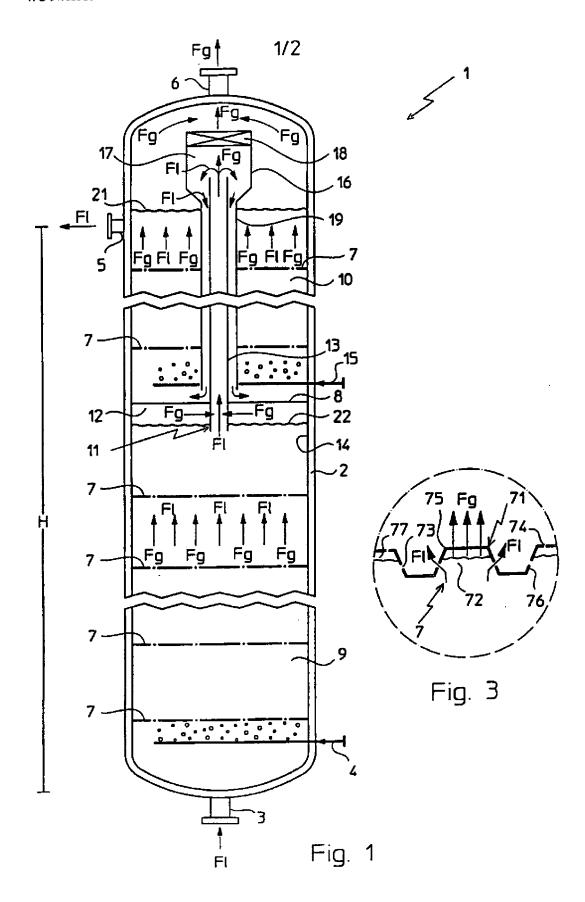
shell (2).

10

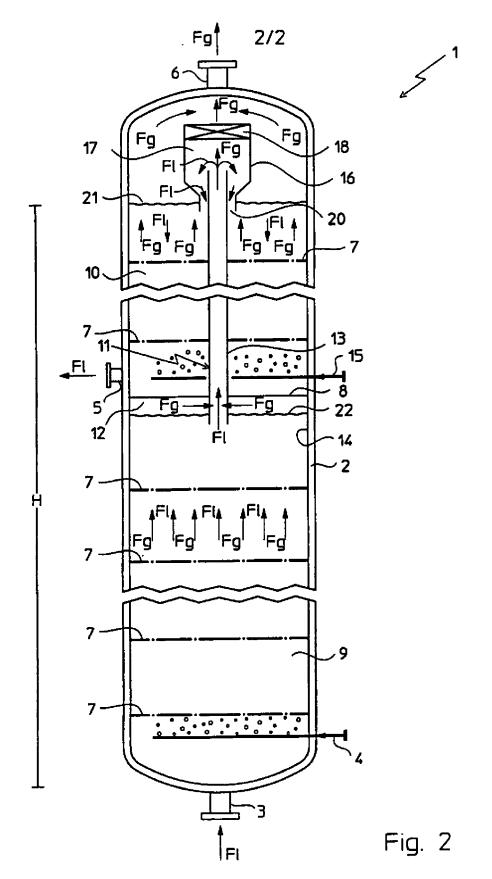
- 10. Reactor according to claim 9, characterized in that said dividing baffle (8) is arranged in said shell (2) at a height between 65% and 75% of the useful height (H) of the shell (2).
- 11. Reactor according to claim 8, characterized in that it comprises a gas/liquid separator (16) placed between said extraction duct (13) and said gaseous phase outlet opening (6), for separation of the liquid phase from said two-phase flow.
- 12. Reactor according to claim 11, characterized in that said gas/liquid separator (16) comprises:
- a chamber (17) for separation of the liquid phase from the gaseous phase, coaxial with said extraction duct 13;
- 15 a demister (18) for separation of the residual liquid phase contained in the gaseous phase coming out of said separation chamber (17);
 - a liquid passage (20) for recycling of the liquid phase to the second reaction space (10).
- 20 13. Reactor according to claim 8, characterized in that said perforated plates (7) comprise:
 - a plurality of elements (71) with substantially trapezoid or rectangular cross section, defining within them respective collection zones (77) for said gaseous phase;
- 25 a plurality of openings (73) for passage of said liquid phase and defined in association with a side wall (76) of said elements (71), and
- a plurality of openings (74) for passage of said gaseous phase and defined in an upper wall of said elements (71) in fluid communication with said collection zones (77);

WO 97/15388 PCT/IB96/01084

with said openings (73) for passage of the liquid phase having a size greater than that of said openings (74) for passage of the gaseous phase.



SUBSTITUTE SHEET (RULE 26)



SUBSTITUTE SHEET (RULE 26)

INTERNATIONAL SEARCH REPORT

Inter nal Application No PCT/IB 96/01004

| A. CLASS IPC 6 | IFICATION OF SUBJECT MATTER B01J10/00 C02F1/02 C07C273 | /04 | |
|--------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------|
| According t | to International Patent Classification (IPC) or to both national class | ification and IPC | |
| | SEARCHED | | |
| Minimum d IPC 6 | locumentation searched (classification system followed by classifica B01J C02F C07C | aon symbols) | |
| Documenta | tion searched other than maramum documentation to the extent that | such documents are included in the fields so | zarched |
| Electronic | iata base consulted during the international search (name of data ba | se and, where practical, search terms used) | |
| C. DOCUM | MENTS CONSIDERED TO BE RELEVANT | | |
| Category * | Citation of document, with indication, where appropriate, of the | relevant passages | Relevant to claim No. |
| A | EP,A,0 538 848 (UREA CASALE S.A. 1993 see the whole document |) 28 April | 1,8 |
| A | EP,A,0 612 560 (W. BIZZOTTO) 31 1994 see the whole document | August | 1,8 |
| ļ | | | |
| A | US,A,4 341 640 (STANDARD OIL COM July 1982 see the whole document | PANY) 27 | 1,8 |
| A | PATENT ABSTRACTS OF JAPAN vol. 13, no. 21 (C-560), 18 Janu & JP,A,63 224785 (TOYO ENG. CORP see abstract; figure | ary 1989 .) | 1,8 |
| | | -/ | |
| | | | |
| χ Fur | ther documents are listed in the combination of box C. | X Patent family members are listed | in annex. |
| 'A' docum | ategories of cited documents nent defining the general state of the art which is not | "I" later document published after the into or priority date and not in conflict to cited to understand the principle or the | th the application but |
| "E" earlier filing | nest which may throw doubts on priority claim(s) or | "X" document of particular relevance; the cannot be considered novel or cannot involve an inventive step when the do | be considered to |
| which cutation "O" docum | t is cited to establish the publication date of another on or other special reason (as specified) nent referring to an oral disclosure, use, exhibition or | "Y" document of particular relevance; the cannot be considered to involve an in document is combined with one or ments, such combination being obvious | ventive step when the ore other such docu- |
| P docum | means sent published prior to the international filing date but than the priority date claimed | in the art. "A" document member of the same patent | |
| | actual completion of the international search | Date of mailing of the international se | arch report |
| 1 | 14 January 1997 | 2 2. 01. 97 | |
| Name and | mailing address of the ISA European Patent Office, P.B. 5818 Patendaan 2 | Authorized officer | |
| | NL - 2220 HV Rijswijk Tgl. (+31-70) 340-2040, Tg. 31 653 epo nl, Eng. (+31 20) 340-3016 | Stevnsborg, N | |

INTERNATIONAL SEARCH REPORT Inferr al Application No

Interr al Application No PCT/IB 96/01004

| | tion) DOCUMENTS CONSIDERED TO BE RELEVANT | PC1/1B 30/01004 |
|---------------------------|-------------------------------------------------------------------------------------------------------------|-----------------------|
| C.(Continua Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
| A | GB,A,911 321 (SHELL INTERNATIONAL RESEARCH MAATSCHAPPIJ N.V.) 21 November 1962 see the whole document | 4-7,11, 12 |
| A | US,A,3 617 033 (Y.ICHIKAWA ET AL.) 2 November 1971 | |
| | | |
| | | |
| | · | |
| : | | |

INTERNATIONAL SEARCH REPORT Inter that Application No.

aformation on patent family members

Inter that Application No PCI/IB 96/01004

| Patent document cited in search report | Publication date | Patent family member(s) | | Publication date |
|-------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------|------------------------------------------------------|----------------------------------------------------------|
| EP-A-0538848 | | CA-A- CN-A- RU-C- US-A- | 2081294 1073152 2056408 5399755 | 24-04-93 16-06-93 20-03-96 21-03-95 |
| EP-A-0612560 | 31-08-94 | | 132392 69400047 69400047 2084518 5486339 | 15-01-96 15-02-96 29-08-96 01-05-96 23-01-96 |
| US-A-4341640 | 27-07-82 | NONE | | |
| GB-A-911321 | ~ ~ = = ~ | NONE | | |
| US-A-3617033 | 02-11-71 | NONE | | |